Mechanical Degradation of Tungsten Alloys at Extreme Temperatures in Vacuum and Oxidation Atmospheres

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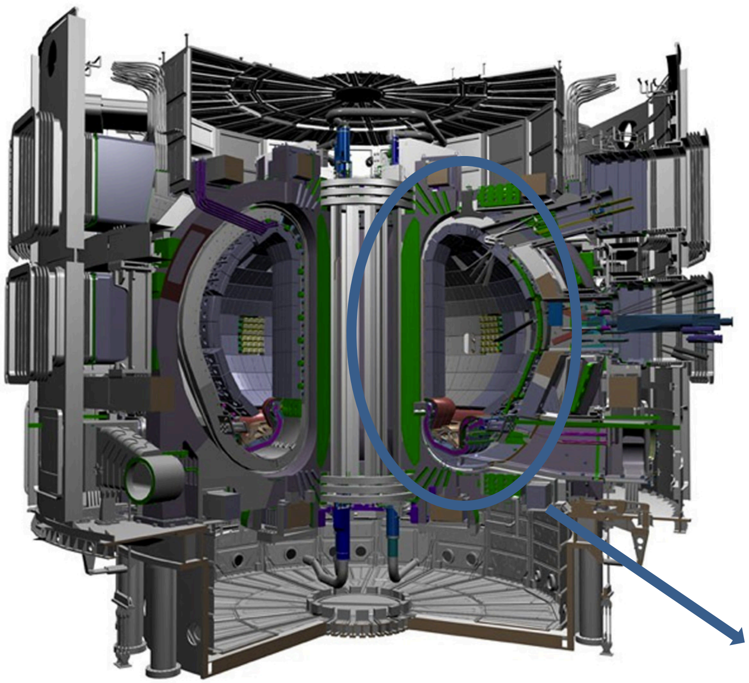
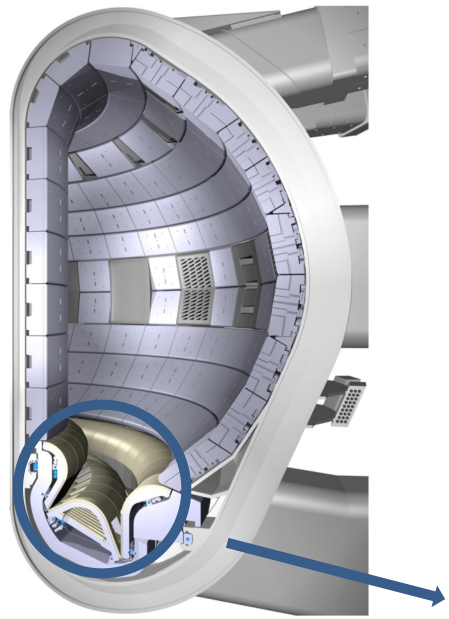
motivation

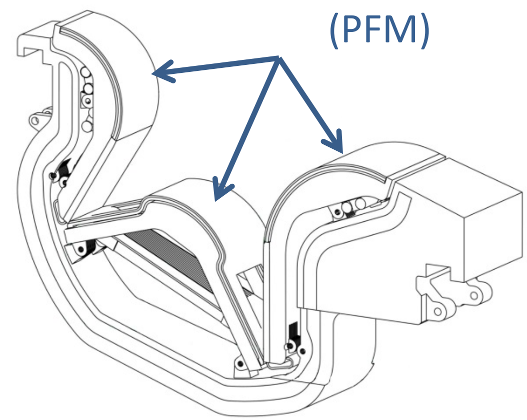


fusion reactor



vacuum vessel

Plasma Facing Material (PFM)



divertor

Extreme conditions

- High ion/neutron flux
- High heat load (~ 10 MW/m^2)
- High temperature
- Thermal stresses & cycling
- Off normal events (e.g. plasma disruption)

why tungsten as PFM?

features an **unique property combination**

- the highest melting point of all metals
- good thermal conductivity
- low tritium retention
- low physical sputtering yield
- high thermal resistance
- high temperature strength

at the divertor perform also a structural application, so ductility is needed and W has a **brittleness** problem
oxidation resistance

Operation temperature range

- Lower limit: ductile-brittle transition temperature (DBTT) 400-650 °C
- Upper limit: rxx temperature (RT) 1300 °C

rxx processes and irradiation effects during operation can induce embrittlement

+ Y_2O_3 + V + Ti + La_2O_3

W – 1 wt% Y_2O_3 (W-1Y)
W – 2 wt% Ti – 1 wt% La_2O_3 (W-2T1L)

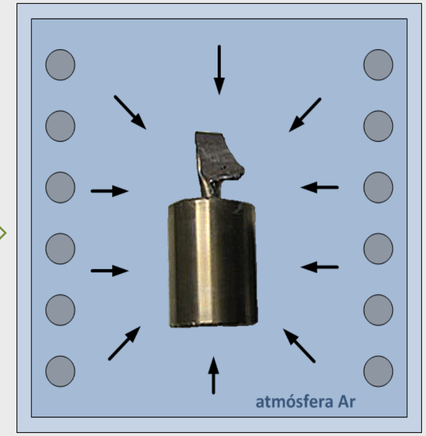
material processed

W	99.9 %	< 5 µm
Ti	99.9 %	< 110 µm
La_2O_3	99.5 %	0.01-0.05 µm
Y_2O_3	99.5 %	0.01-0.05 µm

mix, 4 h

MA
20h

canned
degassed
400 ° C, 24 h

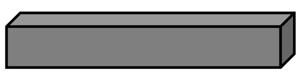


HIP
1300 ° C, 2 h, 195 MPa

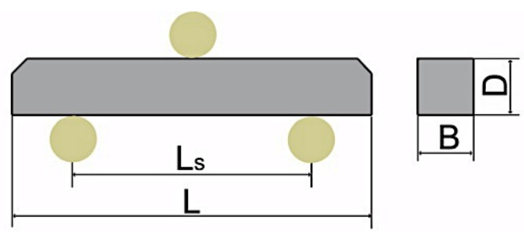
produced in UC3M, Spain

Ø = 30 mm
L = 50 mm

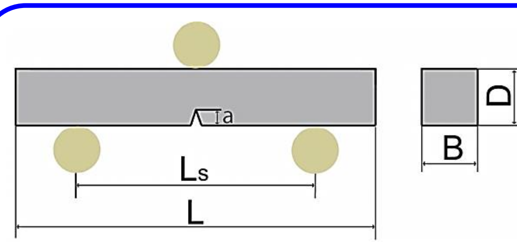
TPB tests samples



1.6 x 1.6 x 25 mm^3



smooth bend bars

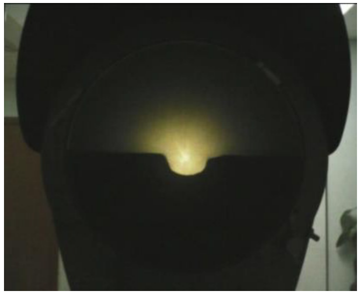


HOW?

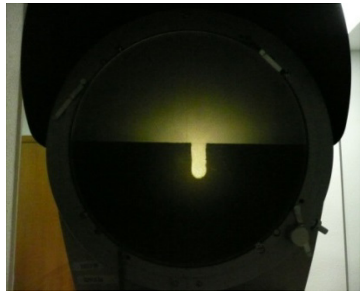
notched bend bars

L = 25 mm
B = 1.6 mm
D = 1.6 mm
L_s = 16 mm
a ~ 400 µm

notched samples

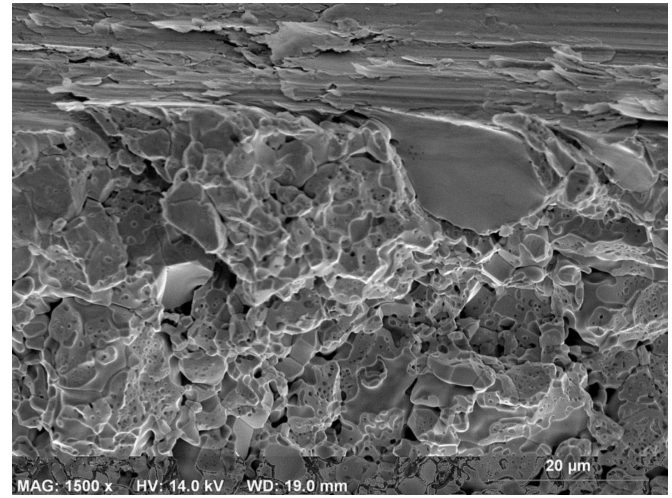


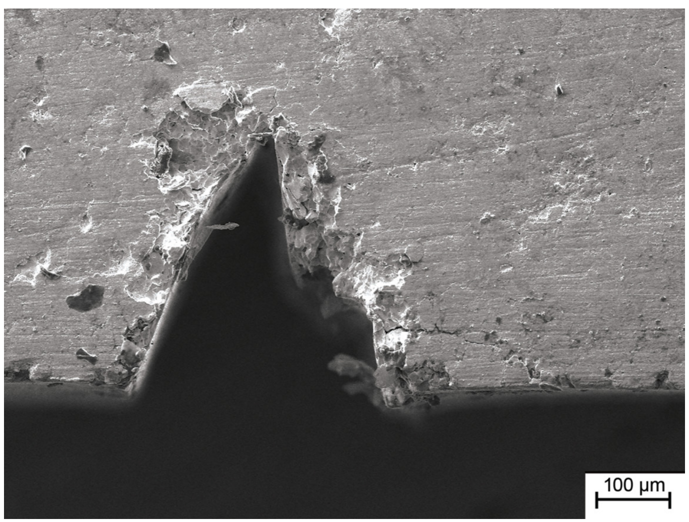
diamond disc
~ 200 µm

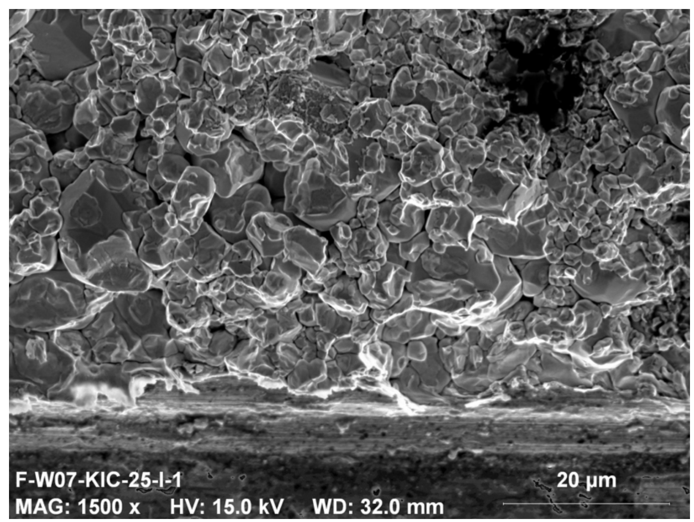


diamond wire
~ 75 µm

fast, plastic damage, big notch root radius







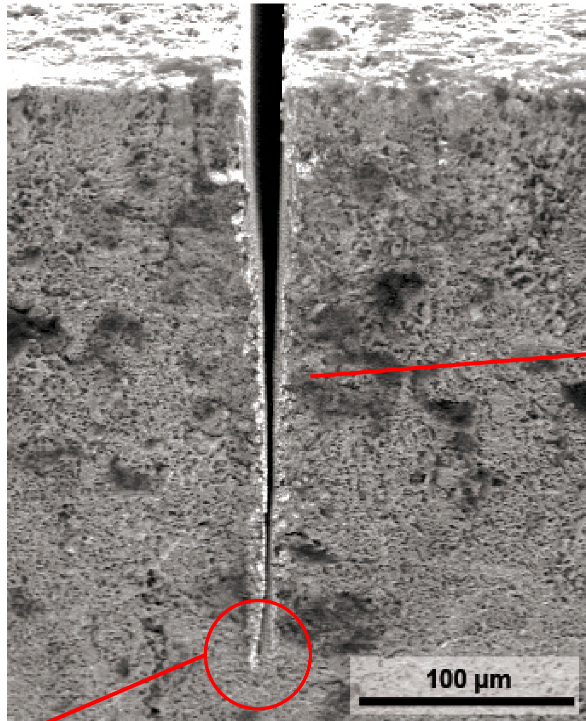
metal blade
~ 5-7 µm

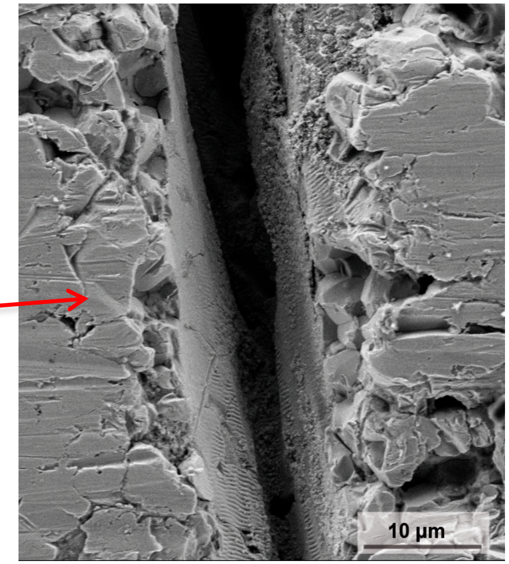
- no plastic damage
- slow complex process

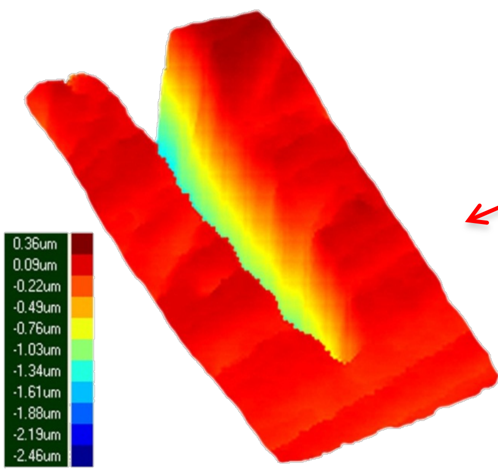
notched samples

laser
1-50 nm

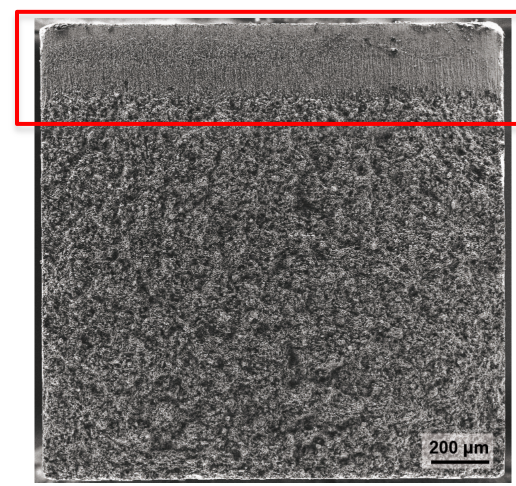
- no plastic damage
- no rxx of the tip grains
- similar to a crack







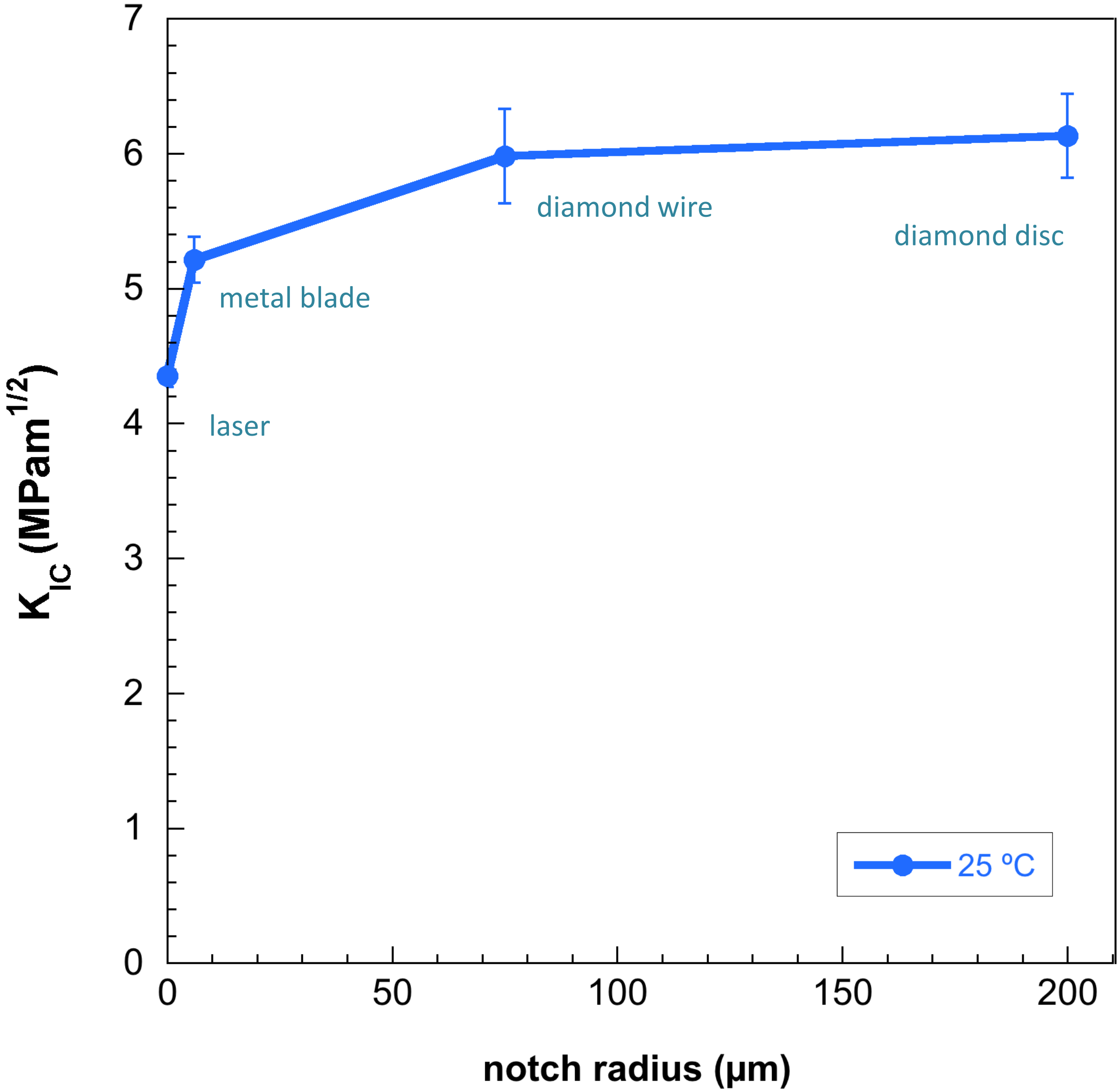
Profilometer image



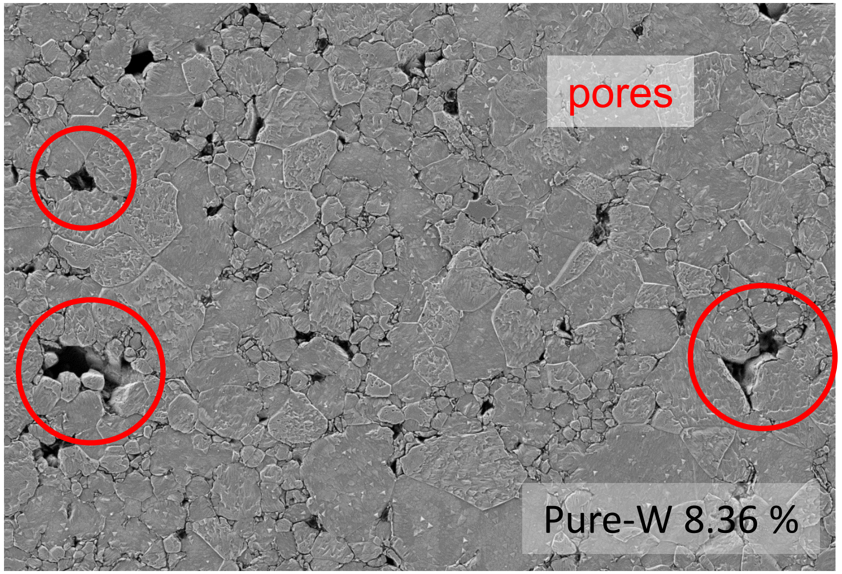
notched samples

TPB tests were performed in notched samples of the 4 methods to check the notch root radius effect

↓ dispersion results
↓ 25-30% of the value

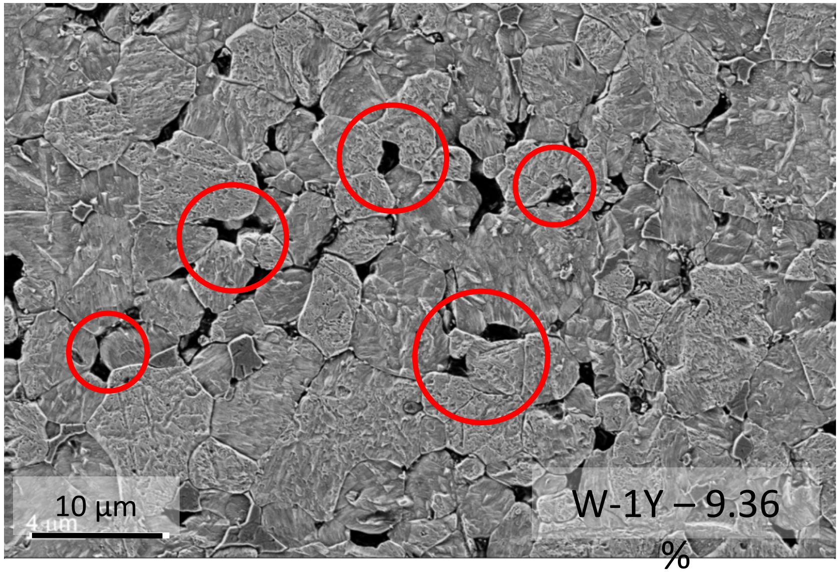


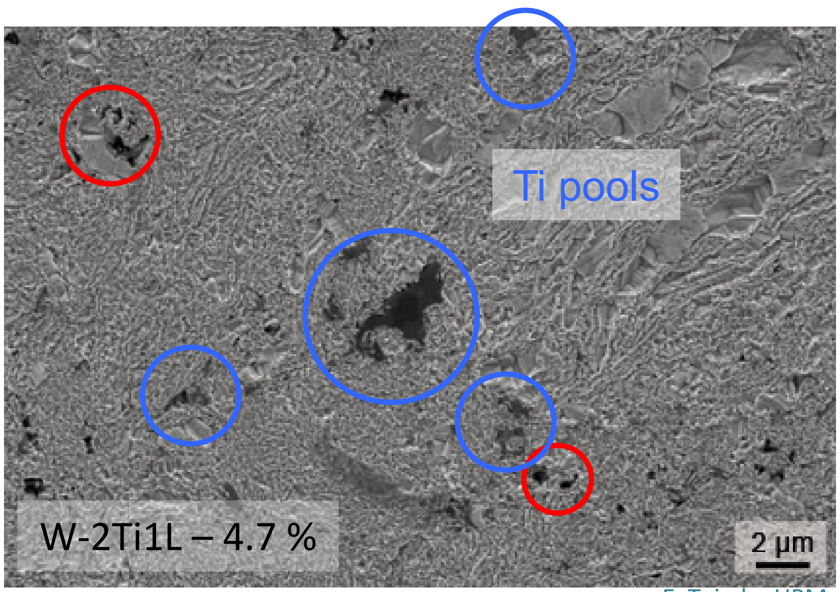
microstructure



W-2T1L:

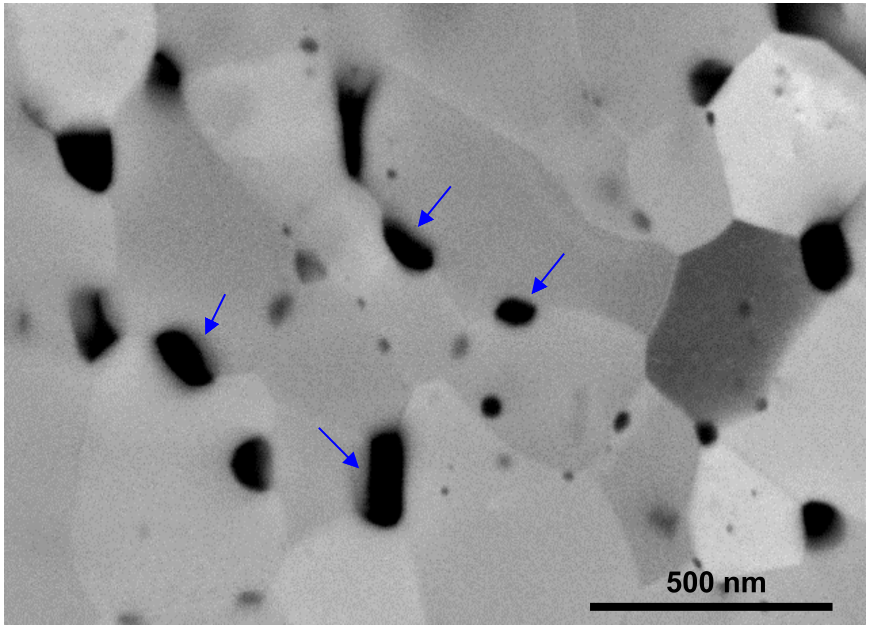
- with the addition of the alloying elements the grain size become nanometric
- change from polyedral coarse grains (pure-W) to coarse W grains and Ti pools surrounded by a W-Ti-La solid solution

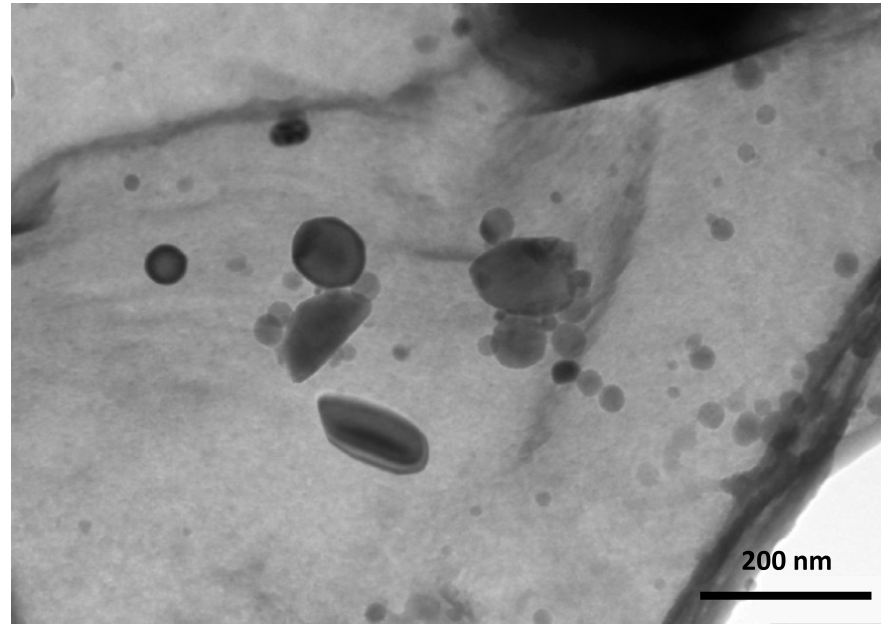




E. Tejado, UPM

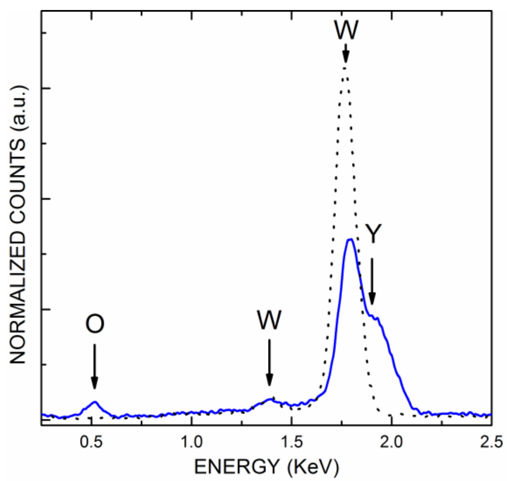
microstructure (W-1Y)





C. Ballesteros, UC3M

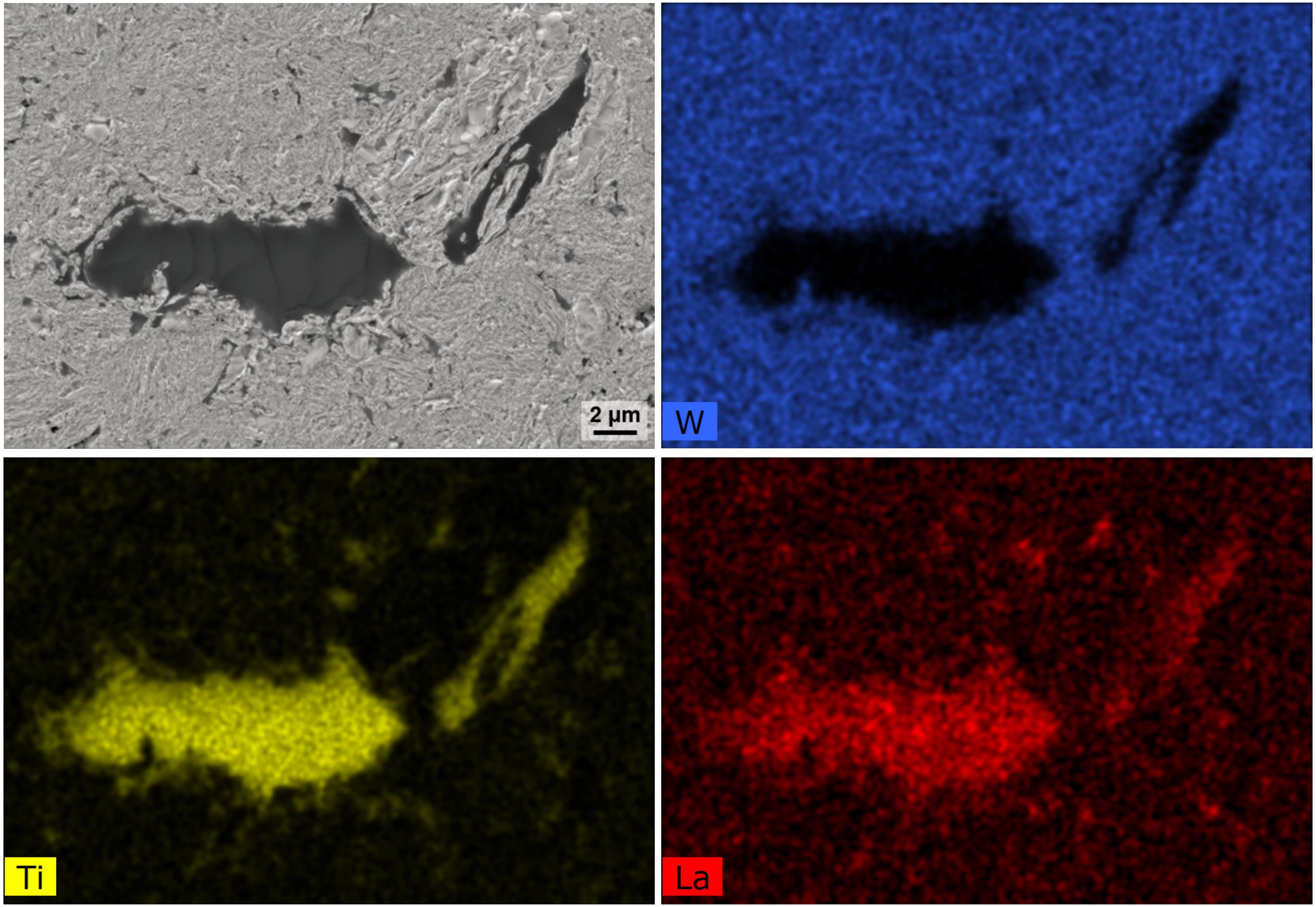
edx spectra from black contrast areas



Y_2O_3 nanoparticles:

- spheroidal dispersoids with variable size
- located in the grain boundaries
- sometimes agglomeration

microstructure (W-2T1L)

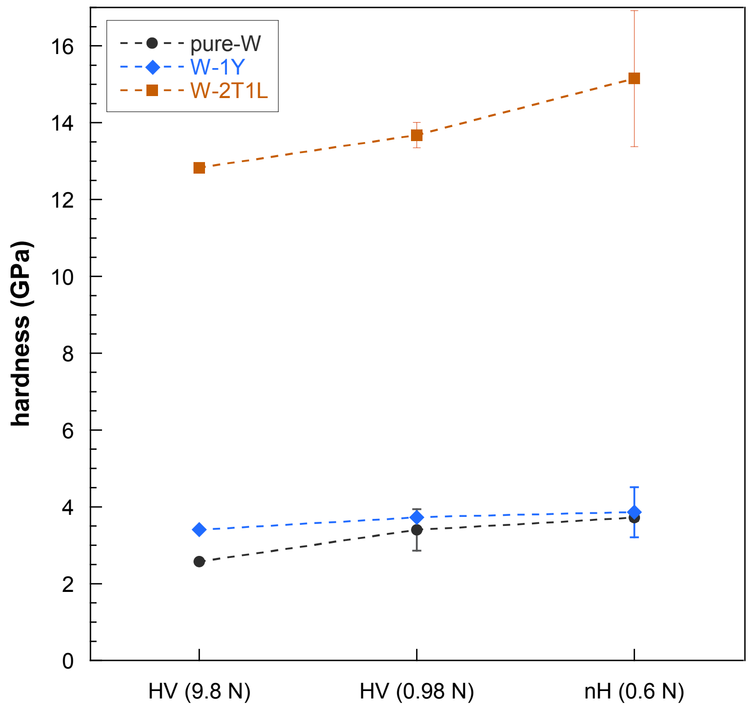


E. Tejado, UPM

FESEM image of W-2Ti-1La_2O_3 alloy with mapping (W), mapping (Ti) and mapping (La).

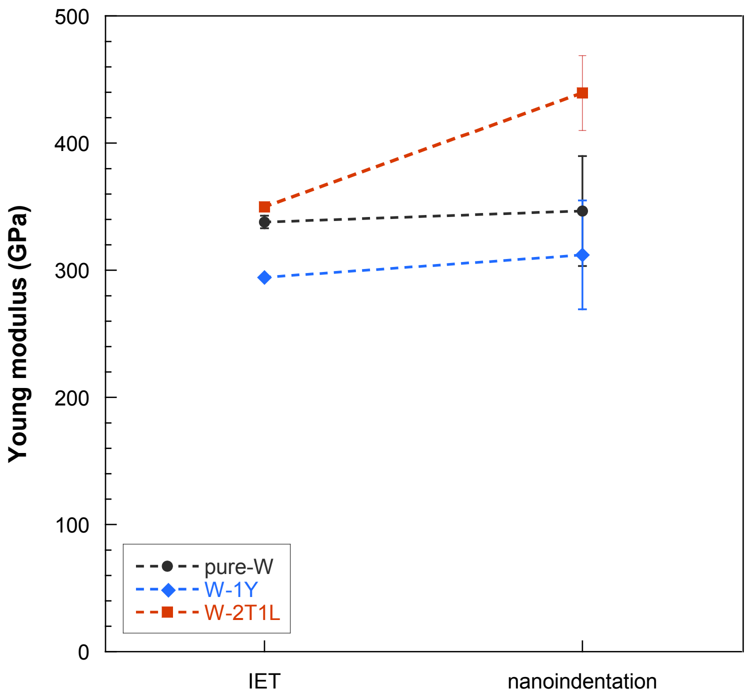
mechanical properties

hardness



small influence of the load
comparable results with instrumented method
W-2T1L ↑ hardness

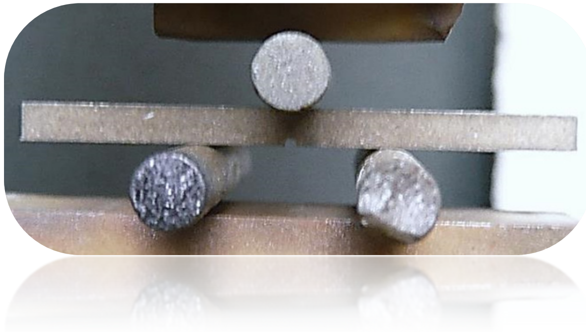
Young modulus



comparable values
W-1Y ↓ E
W-2T-1L Small increase of the values

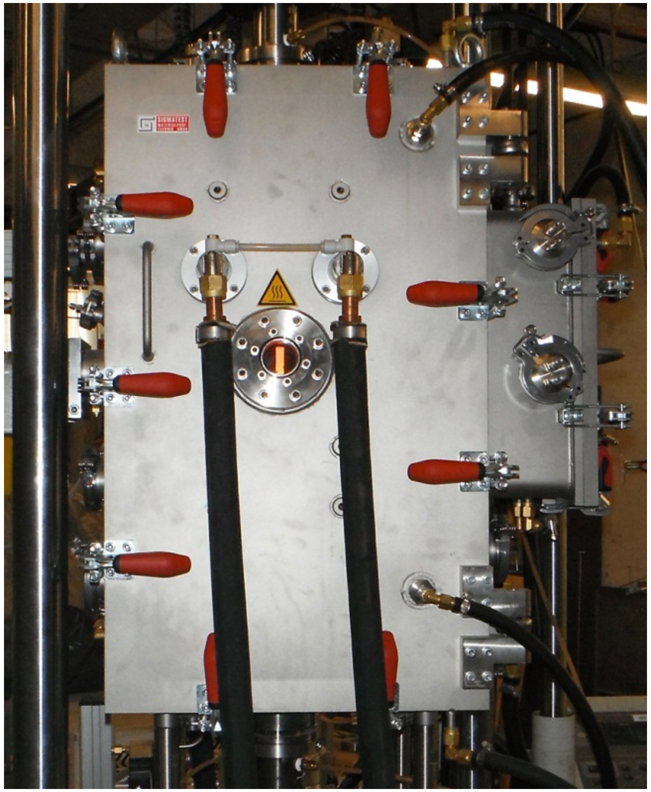
*IET= Impulse Excitation Technique

TPB tests

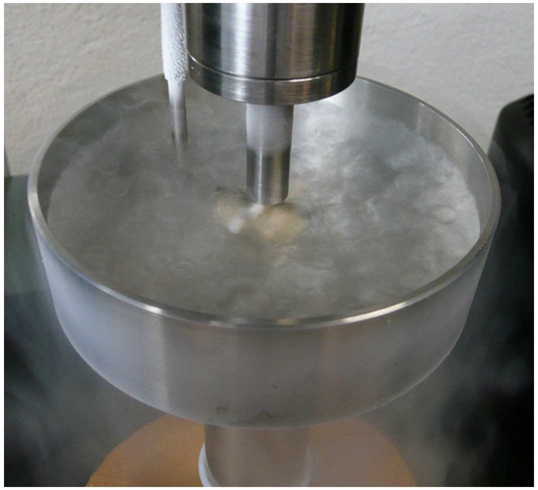


displacement rate:
100 µm/min
heating rate:
50 ° C/min

inert atmosphere (vacuum)

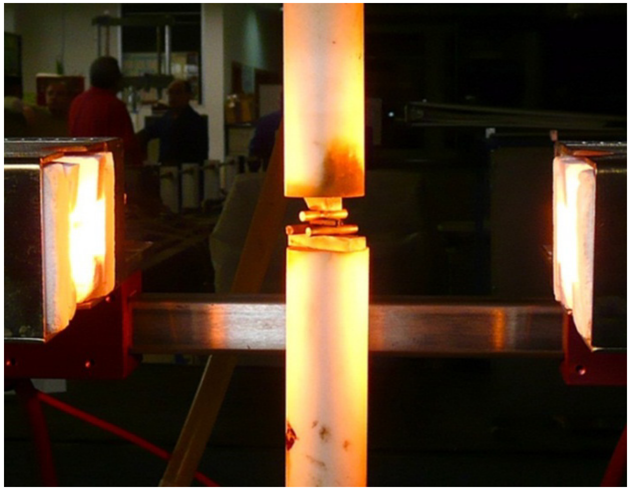


liquid N immersion



T = -196 ° C

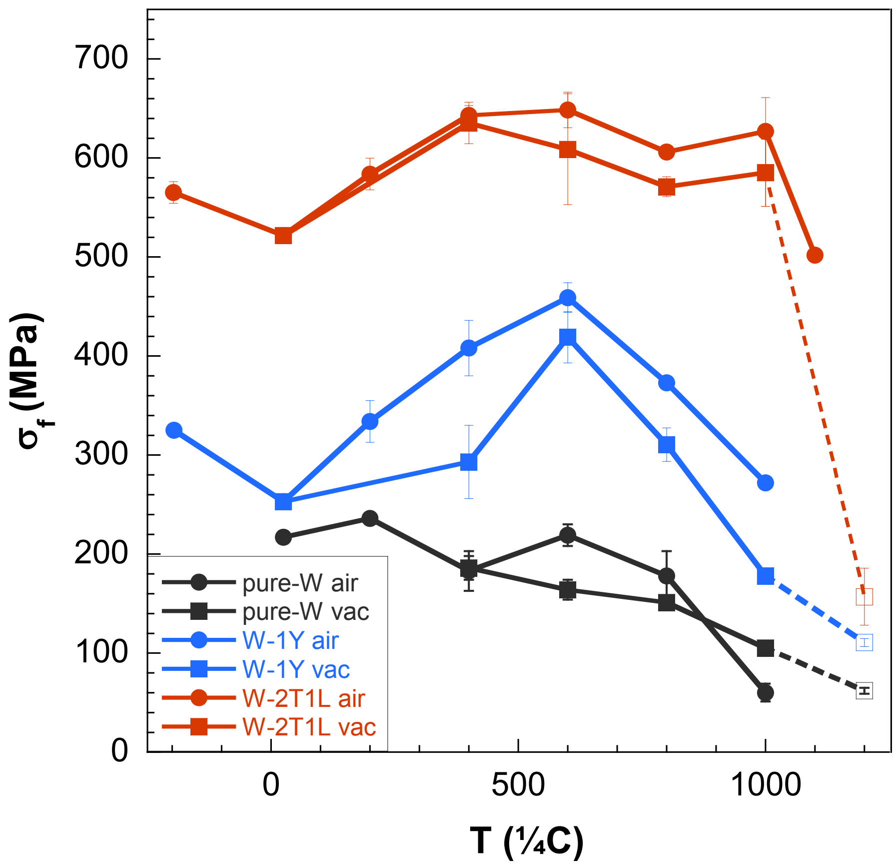
oxidation atmosphere (air)

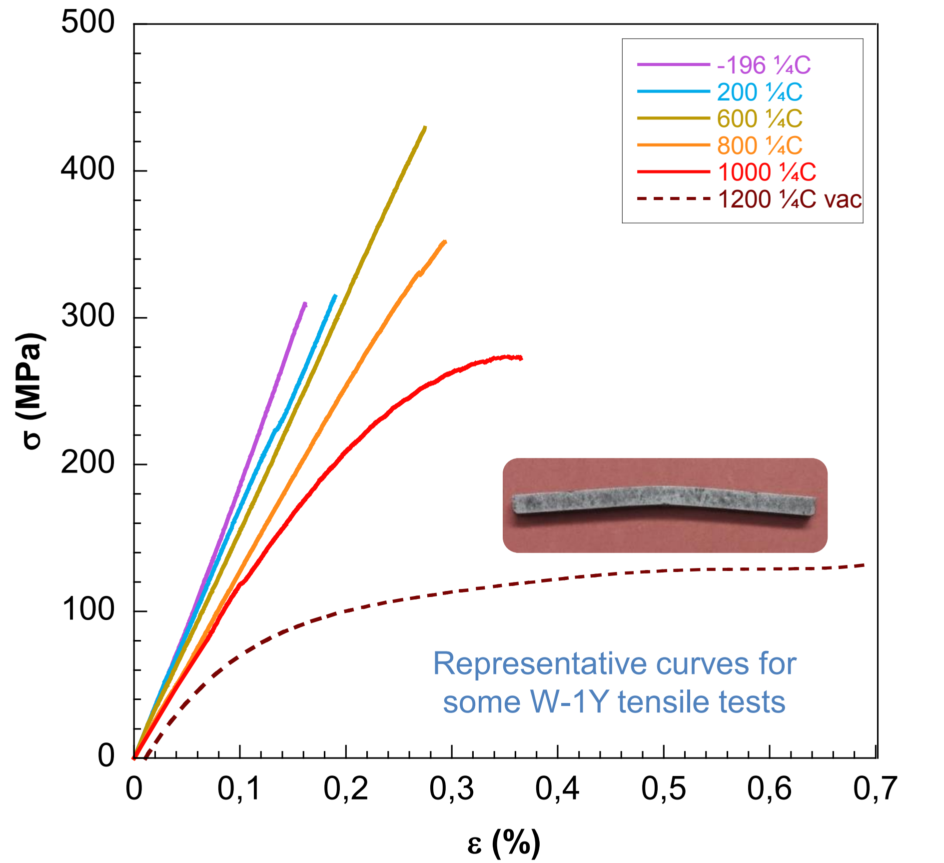


T ≤ 1000 ° C

T ≤ 1200 ° C; 10^{-6} mbar

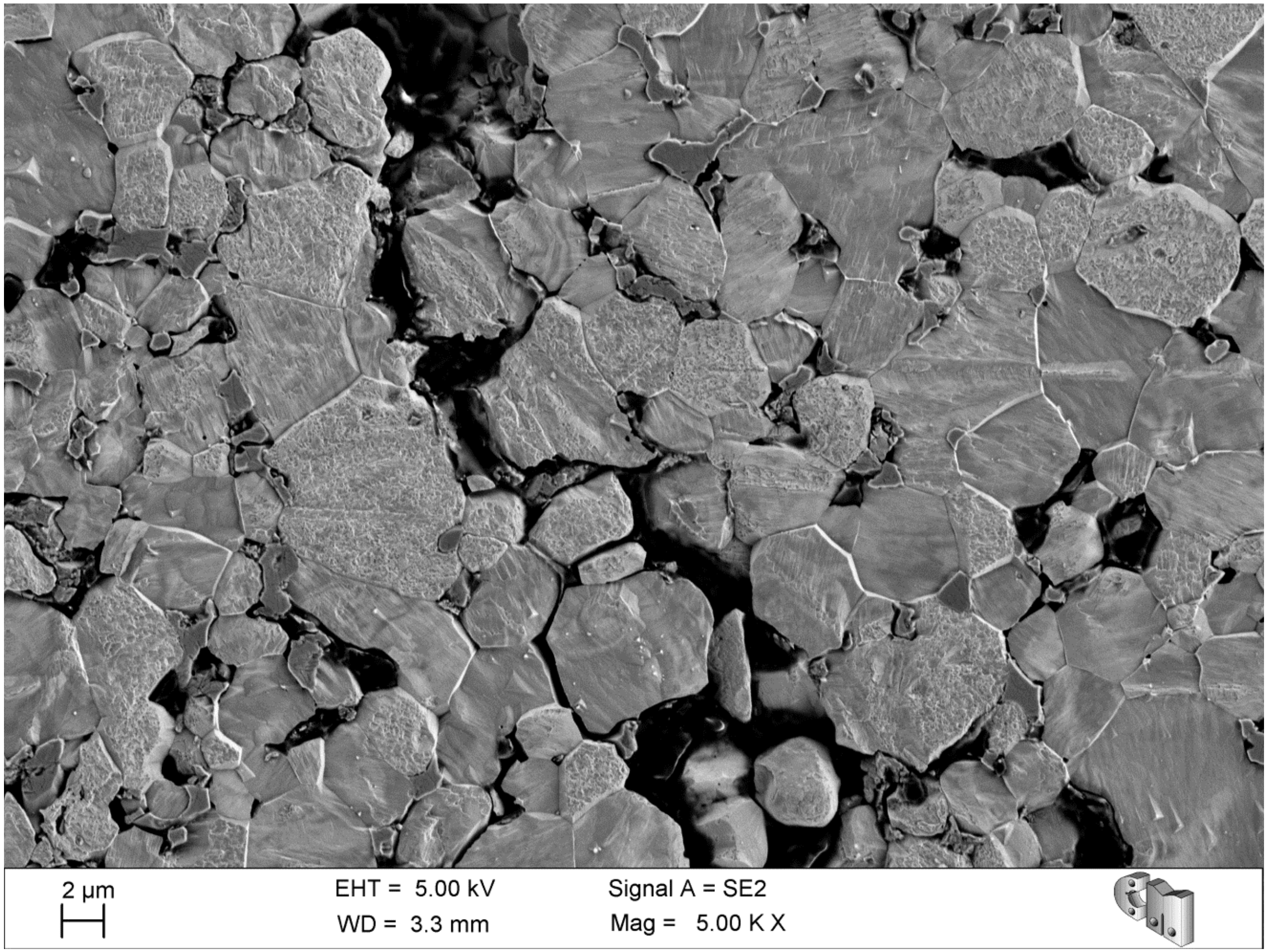
TPB tests – flexural strength

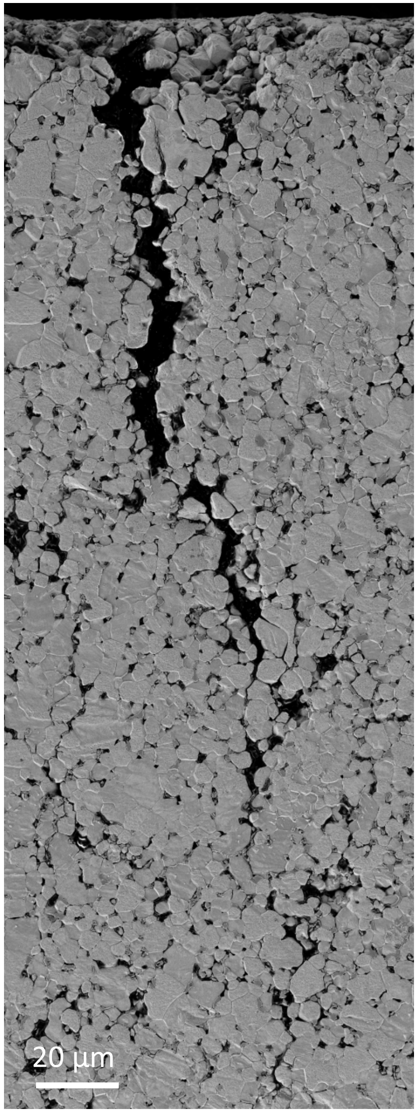




linear elastic until failure (except 1200 ° C)
slightly influence of the atmosphere
↑ flexural strength values (W-2T1L)
thermal degradation – W-2T1L T>1000 ° C

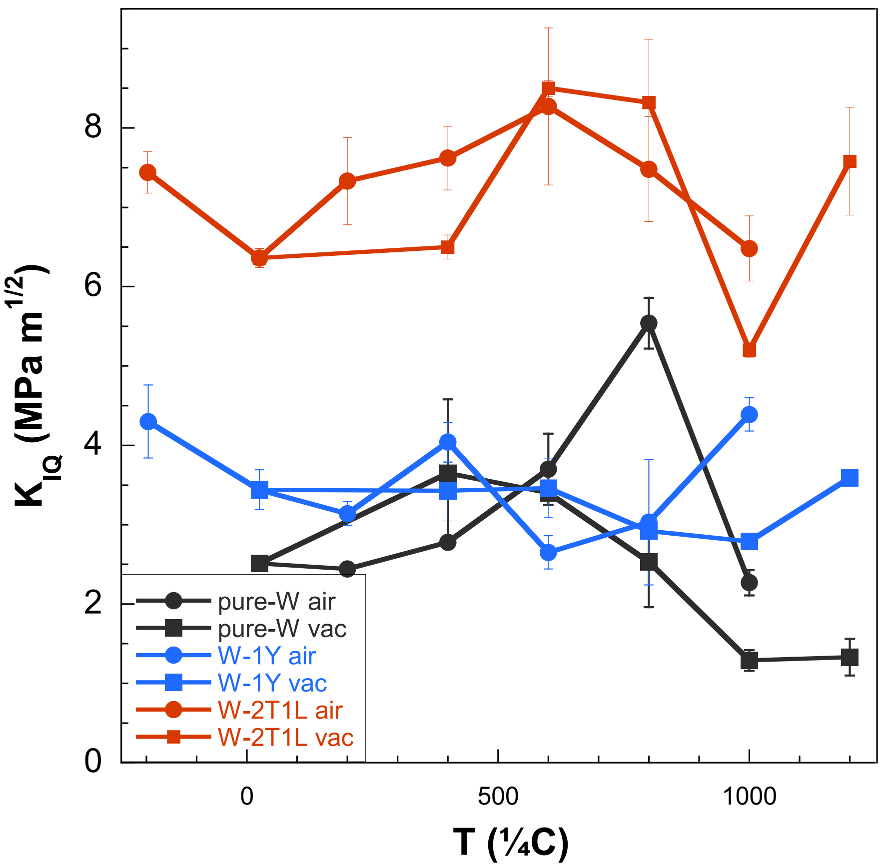
TPB tests – flexural strength

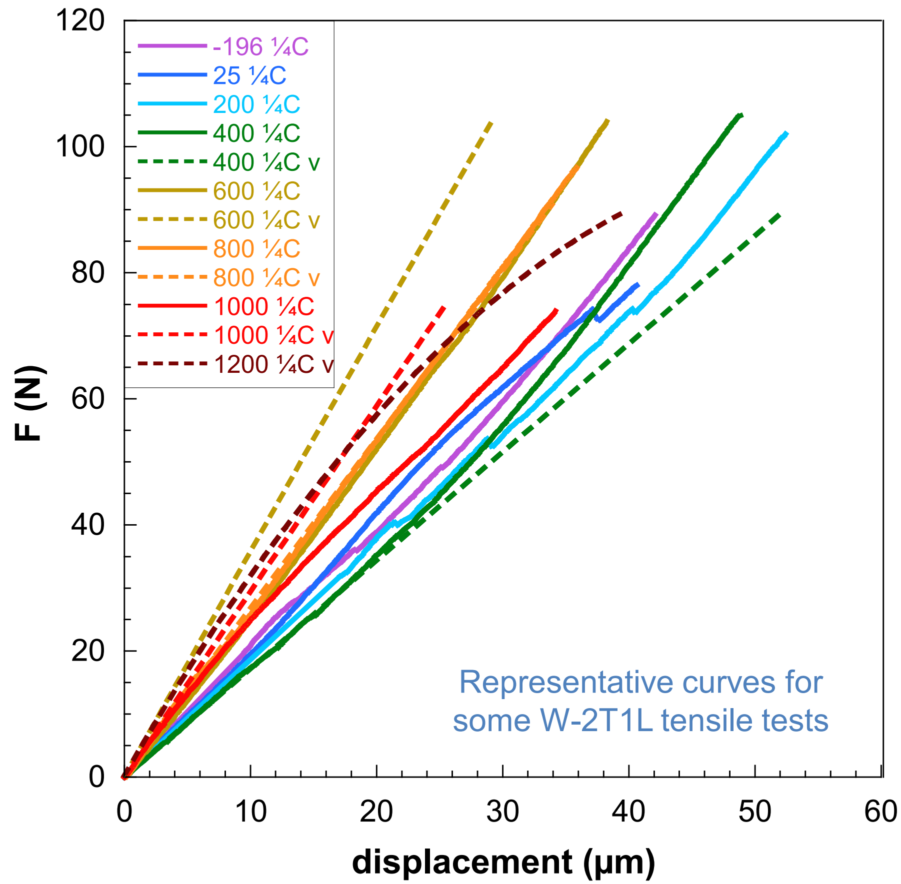




sample tested a 1200 ° C (not break)
intergranular progress of the crack

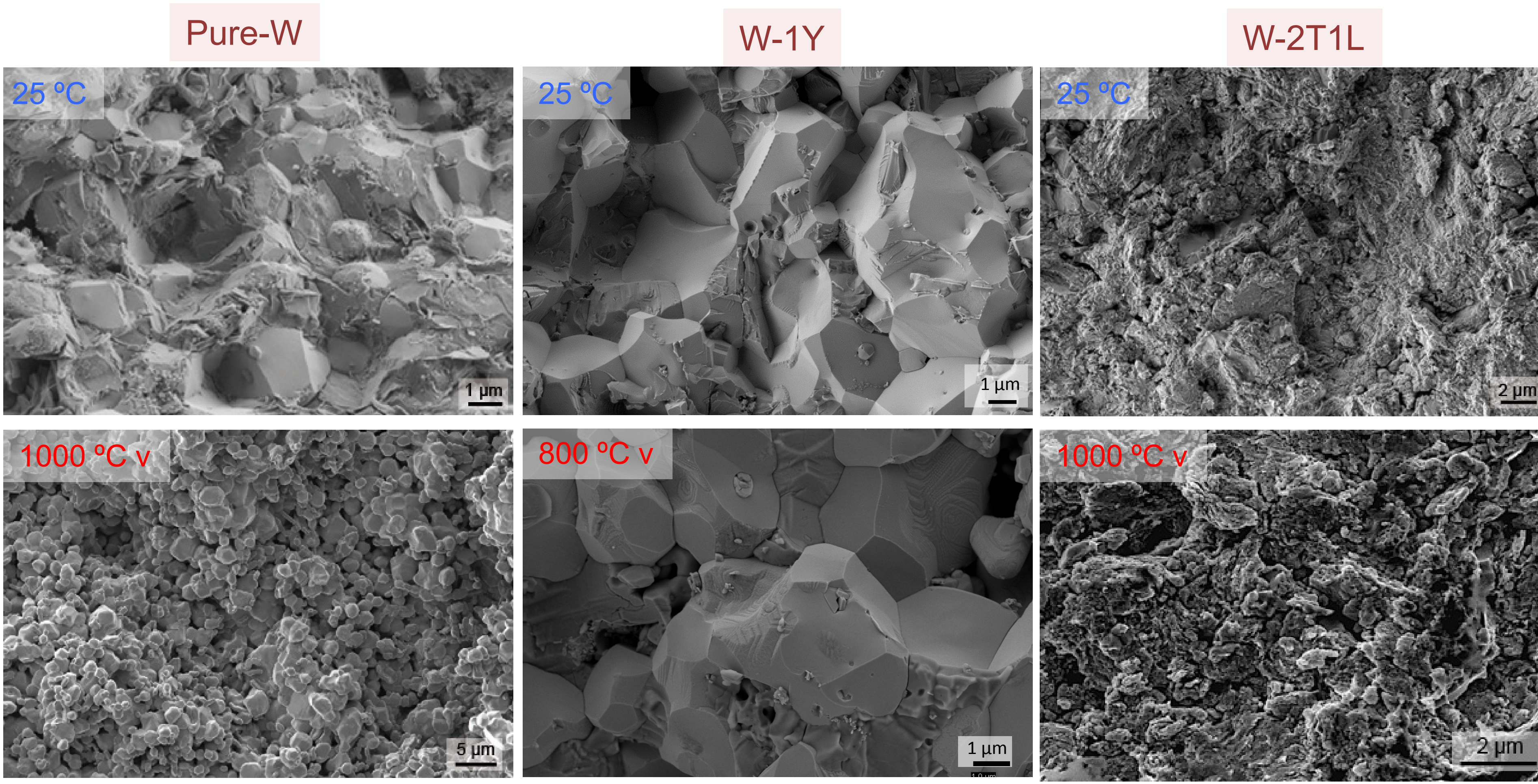
TPB tests- fracture toughness





linear elastic until failure (all the T range)
improve of the behavior: ↑T (W-1Y), all T (W-2T1L)

fracture surfaces

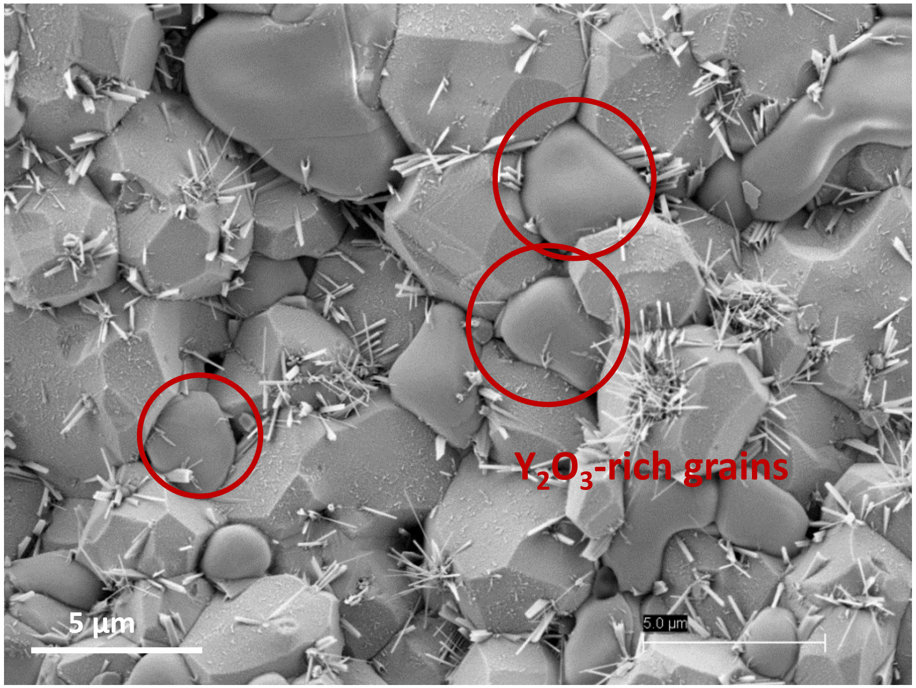


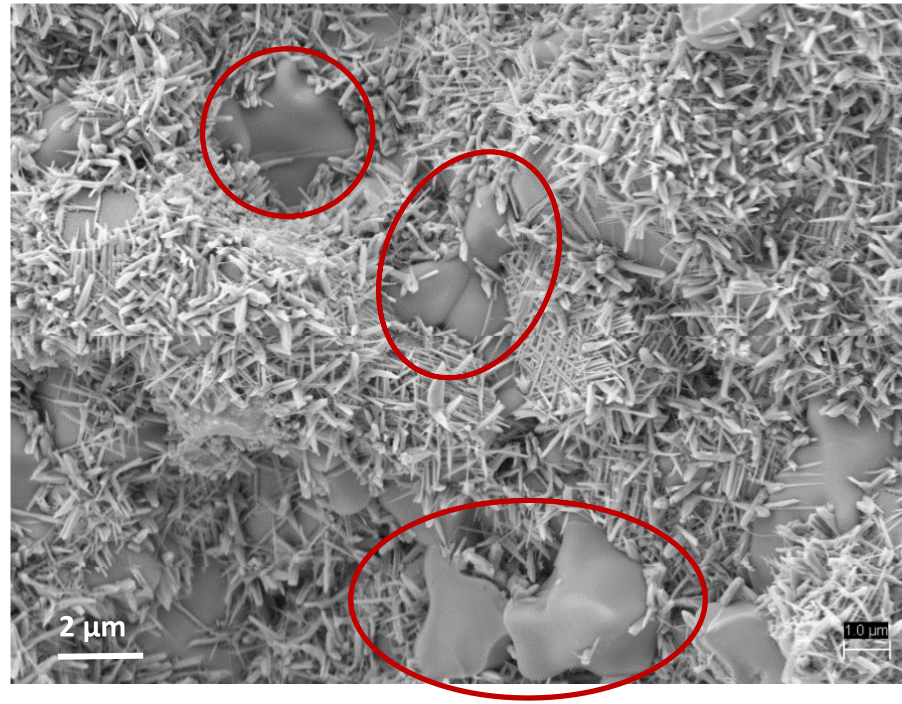
Fracture surfaces of the tested samples at low (up) and high (down) temperature

flat, fracture decohesion by grain boundary

fracture surfaces

W-1Y 1000 ° C, air





Bimodal morphology of grains:
- polyhedral coarse grains of W (surrounded by oxide needles)
- rounded smaller grains with dispersion of Yttria nanoparticles

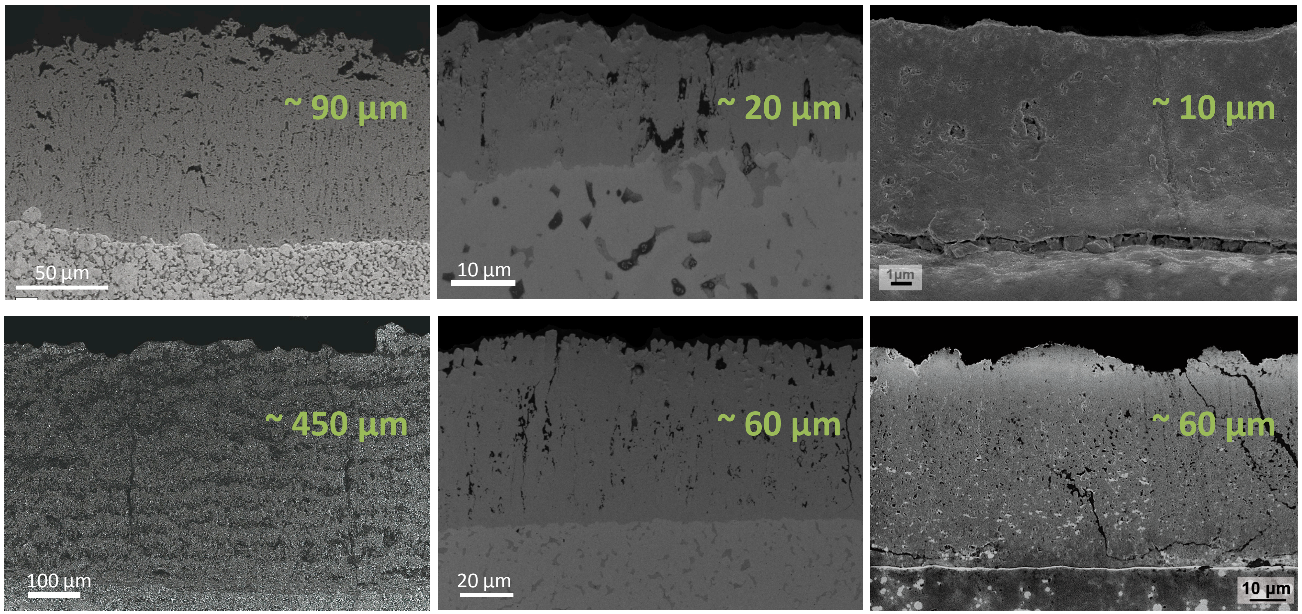
outlook

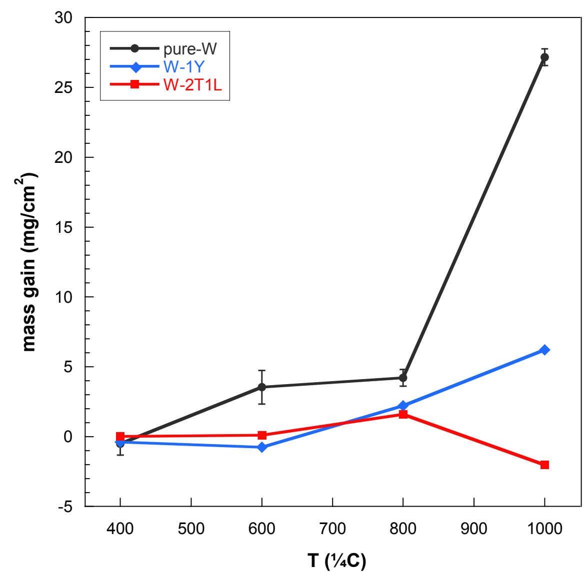
- Powder route was used to process W-alloys with additions of Y_2O_3, Ti and La_2O_3.
- TPB tests were perform to obtain flexural strength, yield strength and fracture toughness (-196 to 1200 ° C, air & vacuum). Moreover nanoindentation, Vickers and IET tests (H, E).
- Laser notching of the samples show a notch like crack with a root radio between 1-50 nm and less dispersion of the results.
- W-1Y_2O_3:
- Slightly enhancement of properties at high T, but increased porosity.
- Microstructure - Yttria nanoparticles located in the grain boundaries and bimodal morphology of grains.
- Fracture analysis - intergranular breakage between grain boundaries (support the brittle behavior of the performed tests up to high T).
- W-2Ti-1La_2O_3:
- The porosity slightly decreases and the grain size became nanostructured.
- Mechanical properties according to TPB tests increase. The values remain stables in all the temperature range (exc. 1200 ° C, ductile).
- Fracture surfaces remain flat and show intergranular breakage between grain boundaries. Supported by brittle behavior of the performed TPB tests.

Thank you for your attention!

oxidation

Pure-W | W-1Y | W-2T1L





Cross sections of the scales developed in the samples oxidized in air after 15 min exposure at 800 (up) and 1000 ° C (down)

- Pure-W is highly reactive with oxygen above 400 ° C, but with the addition of the alloying elements (Ti, La_2O_3), the oxidation processes decreased (W-1Y, W-2T1L).
- The outer yellow scale remain thinner and the mass gain is lower in all the temperature range
- At 1000 ° C some cracks appears in the scale as a consequence of the stresses produced during oxide growing.